

Attorney's Docket No.: 00169-027001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Michael D. Gilbert Art Unit : 1771
Serial No. : 09/352,976 Examiner : Victor S. Chang
Filed : July 14, 1999
Title : **ELECTRICALLY DISBONDING MATERIALS**

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION OF A.C. MAKRIDES UNDER 37 C.F.R. § 1.132

I, Alkis C. Makrides, declare as follows:

1. I am a citizen of the United States and currently live at 50 Grafton Street, Newton, MA 02459.

2. I received a Bachelor's degree in Chemistry from the University of Texas (Austin) in 1951 and a Ph.D. in Physical Chemistry from the University of Texas (Austin) in 1957.

3. I was employed by Union Carbide Metals Company (Niagara Falls) as Research Chemist from 1957-1959; by Mobil Oil Company (Dallas) as Senior Research Chemist from 1960 to 1962; by Tyco Laboratories, Inc. (now Tyco International) as Group Leader, Associate Director of Research, Director of Research, and Vice President from 1962 to 1970; by EIC Laboratories, Inc. as President and Chief Executive Officer from 1971 to present.

4. I have conducted research in Electrochemistry, Batteries, Surface Chemistry (Adsorption and Catalysis), and Corrosion for over twenty years.

5. I have been employed by EIC Laboratories, Inc. since 1971, and my current position is Chief Executive Officer and my responsibilities include the overall direction of the research work at EIC Laboratories.

6. A list of my articles, patents, and patent applications is attached.

Applicant : Michael D. Gilbert
Serial No. : 09/352,976
Filed : July 14, 1999
Page : 2 of 4

Attorney's Docket No.: 00169-027001

7. I have examined and reviewed Moulton et al., U.S. Patent No. 5,441,830 ("Moulton"), and Koga et al., U.S. Patent No. 5,565,284 ("Koga") in detail. I have also examined and reviewed the Office Action dated 03/22/2005.

8. A fundamental fact of all electrochemical systems is that the interface between two electronically conducting phases cannot support an electrochemical (or faradaic) reaction. This fundamental fact is reflected in the requirement that an ionic electrolyte is a necessary component of all electrochemical devices (batteries, fuel cells, etc.). A potential gradient between electronic conductors causes a flow of electrical (electronic) current; a voltage gradient between an electronic conductor and an ionic conductor may cause a chemical reaction.

9. The assertion that Moulton's composite electrode discloses on the claimed invention is flawed because Moulton's composite electrode – or any other composite or solid electrode – is by necessity an electronic conductor. If it is not, then it is not a useful "electrode." As an electronic conductor, it cannot support a faradaic reaction at the electrode/current collector interface. In fact, if such a reaction were possible, the device incorporating such an electrode would fail, probably catastrophically.

10. The Examiner notes that "Moulton's teaching of methods for enhancing the adhesion of composite electrodes onto conductive foils implicitly teaches that while the bond can be enhanced, eventually it can be disbonded, i.e. disbondable". This argument is not germane to the claimed invention. Any bond can eventually be disbonded in some fashion – for example, by applying sufficient force on the interface. The invention claims an easy, quick, and reliable method, namely the application of a voltage to produce a faradaic reaction at the interface.

Moulton is actually explicit about the modes of disbonding. To quote "when conductive plastic foils are used as the current collector, they are in direct contact with a composite electrode and are susceptible (sic) to swelling due to contact of the current collector with the electrolytic solvent found in the electrode. Such swelling adversely affects the performance of the battery" (Col. 1, lines 50-57). Also, Moulton notes that bonding of a composite electrode to the current

Applicant : Michael D. Gilbert
Serial No. : 09/352,976
Filed : July 14, 1999
Page : 3 of 4

Attorney's Docket No.: 00169-027001

collector may be poor in the first place and notes that "the paste can become dislodged from the foil. In turn, such dislodgement will result in defects in collecting current from that cell" (Col. 1, lines 67-68, Col. 2, lines 1-2). Neither "disbonding" mechanism involves the application of voltage across the interface or a faradaic reaction at the interface.

11. The Examiner notes "that it is old and well known that the bond between a composite electrode and an electrically conductive surface is weakened by a faradaic reaction at the interface, as evidenced by the teaching of Koga which expressly teaches that charge-discharge cycles exacerbates (sic) the interfacial adhesion (bond) between the current collector and the electrode layer (Col. 1, lines 39-52)."

This statement confuses interfaces – Koga does not refer to the interface between "a composite electrode and an electrically conductive surface" or to a faradaic reaction at this interface, which, as we pointed out before, is not possible. Actually, Koga is describing a bulk effect, the expansion and contraction of lithiated and delithiated electrode material which leads to fracture of the material and shedding of "fine particles". As he points out, this is a result of "numerous repetition of charge-discharge cycles" (Col. 1, line 42) and is unrelated to the disbonding described in the present invention which occurs after a single, short application of a voltage across the bond interface.

12. There are a number of other confused statements by Moulton and, derivatively, by the Examiner. For example, Moulton claims in his definition of composite electrodes the inclusion of "an alkali salt" (Col. 8, line 16). This is in fact incorrect – the salt must be a lithium salt; other alkali salts will not do since batteries utilizing alkali metals other than lithium are unknown. On his part, the Examiner notes that V_6O_{13} is "prepared by heating ammonium metavanadate ($NH_4^+VO_3^-$), i.e. an ammonium salt (Col. 18, lines 57-61)." Of course, the purpose of the heating is to drive off ammonia so that the final composition contains no ammonium salt – contrary to the present invention which uses an ammonium salt.

What is crucial is the fundamental difference between, on the one hand, a composite electrode – a structure composed up to 90% of some electrochemically active compound, usually an oxide, which is either intrinsically electronically conductive or can be made so by the addition

Applicant : Michael D. Gilbert
Serial No. : 09/352,976
Filed : July 14, 1999
Page : 4 of 4

Attorney's Docket No.: 00169-027001

of an electronic conductor, usually carbon, held together by a binder and, on the other hand, an adhesive containing an electrolyte which supports an electrochemical (faradaic) reaction at the bonding interface. These two materials are fundamentally different, notwithstanding the inclusion, in varying amounts, of some common ingredients. In short, a composite electrode is not an electrochemically disbondable adhesive, even with the inclusion of binders for holding together its components, since an electrode is necessarily an electronic conductor.

13. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the instant patent application or any patent issuing thereon.

Dated: 6-22-2005

Alkis C. Makrides
Alkis C. Makrides

A.C. MAKRIDES
CEO
EIC LABORATORIES, INC.

Education:

Ph.D., (Physical Chemistry), University of Texas, 1957.
B.A. (Summa Cum Laude), University of Texas, 1951.
Research Fellow, Institute for the Study of Metals, University of Chicago, 1954-55.
Research Fellow, American Petroleum Institute, University of Texas, 1955-57.

Professional Experience:

1971 - Present President and CEO, EIC Laboratories, Inc.

1970 - 1975 Adjunct Professor of Chemistry, Boston College, Boston, MA.
Associate Director, Environmental Center, Boston College,
Boston, MA.

1962 - 1970 Director of Research, Vice President, Tyco Laboratories, Inc.,
(Now Tyco International), Waltham, MA.

1960 - 1962 Senior Research Chemist, Mobil Oil Company, Dallas, TX.

1957 - 1959 Research Chemist, Union Carbide Metals Company, Niagara
Falls, NY.

Professional Societies:

Member of the American Chemical Society, The Electrochemical Society, Faraday Society, American Institute of Chemists, New York Academy of Sciences, National Association of Corrosion Engineers.

Honors:

Scholastic: Phi Beta Kappa, Tau Beta Pi, Phi Lambda Upsilon, Omega Chi
Epsilon, Phi Eta Sigma, Sigma Xi.

Professional: Turner Memorial Award of the Electrochemical Society, 1957.
Young Author's Prize of The Electrochemical Society, 1960.

Accomplishments:

Dr. Makrides has carried out and directed research and development in adsorption, catalysis, chemical kinetics, electrochemical kinetics, thermochemistry, batteries, and corrosion. Dr. Makrides is the author of numerous reports on government sponsored research and development, and a number of patents.

Publications:**Adsorption and Catalysis:**

Charge-transfer, No-bond Adsorption, J. Chem. Phys., 22, 1800 (1954).
Adsorption Inhibition of Acid Dissolution of Metals, J. Phys., Chem., 59, 707 (1955).
Heats of Immersion of Silica in Water, J. Phys. Chem., 63, 594 (1959).
Thermal Aging of Silica Gels, J. Phys. Chem., 63, 1789 (1959).
Adsorption and Oxidation of Formic Acid on Platinum Electrodes, J. Phys. Chem., 68, 1448 (1964).
Studies of Electrocatalysis, Proc. 20th Ann. Power Sources Conference, 20, 5 (1966).
Condensation of Silica from Supersaturated Silicic Acid Solutions, J. Coll. Interface Sci., 73, 345 (1980).

Absorption and Diffusion of Hydrogen in Metals:

X-Ray Studies of Hydrogen-Silver-Palladium Alloys, J. Phys. Chem., 68, 2154 (1964).
Absorption of Hydrogen by Silver-Palladium Alloys, J. Phys. Chem., 68, 2160 (1964).
Diffusion of Hydrogen through Palladium and Palladium-Silver Alloys, Trans. Faraday Soc., 61, 932 (1965).
Diffusion of Hydrogen through Palladium, Engl. Ind. Technical Bull., 7, 51 (1966).

Electrochemistry:

Current-Potential Relations for Dissolution of Metals, J. Electrochem. Soc., 102, 363 (1955).
Stoichiometric Numbers and Hydrogen Overpotentials, J. Electrochem. Soc., 104, 677 (1957).
Solid Electrodes for Hydrogen Overpotential Studies, J. Electrochem. Soc., 107, 472 (1960).
Electrode Assembly for Electrochemical Measurements, J. Electrochem. Soc., 107, 782 (1960).
On the Mechanism of the Hydrogen Reaction, J. Electrochem. Soc., 109, 256 (1962).
Hydrogen Overpotentials on Nickel in Alkaline Solution, J. Electrochem. Soc., 109, 977 (1962).
Anodic Dissolution and Oxygen Reduction on Nickel Compounds in Perchlorate Solutions, J. Electrochem. Soc., 111, 278 (1964).
Kinetics of Redox Reactions on Passive Electrodes, J. Electrochem. Soc., 111, 932 (1964).
Kinetics of the Ferric-Ferrous Reaction on Iron-Chromium Electrodes, J. Electrochem. Soc., 111, 400 (1964).
Hydrogen Evolution at Dilute Platinum and Palladium Amalgam Electrodes, Trans. Faraday Soc., 60, 938 (1964).
Surface Oxidation of Gold Electrodes, J. Electrochem. Soc., 111, 1122 (1964).
Hydrogen Evolution at a Dropping Indium Amalgam Electrode, Trans. Faraday Soc., 60, 1664 (1964).
Hydrogen Peroxide Reactions on Gold Electrodes, J. Electrochem. Soc., 112, 756 (1965).
The Electrical Double Layer at Indium Amalgam Electrodes, J. Electroanal. Chem., 9, 237 (1965).
Electrochemistry of Surface Oxides, J. Electrochem. Soc., 113, 1158 (1966).
Rechargeable Li/TiS₂ AA Cells, 34th International Power Sources Symp., IEEE, June 25-28, 1990, p. 167.

Corrosion:

Mechanism of Inhibition by Organic Compounds, Ind. Eng. Chem., 46, 523 (1954).
Effect of Thiourea on Dissolution Rate of Iron, Ind. Eng. Chem., 47, 1773 (1955).
Dissolution of Metals in Aqueous Acid Solutions, J. Electrochem. Soc., 105,
156 (1958).
Some Observations on the Mechanism of Inhibition, J. Electrochem. Soc., 106,
7 (1959).
Inhibition of Metal Dissolution by Ferric Sulfate, J. Electrochem. Soc., 107, 877 (1960).
Dissolution of Iron in Ferric Sulfate Solutions, J. Electrochem. Soc., 107, 869 (1960).
Inhibition by Ferric Sulfate of Dissolution of Stainless Steel, J. Electrochem. Soc.,
108, 412 (1961).
Some Electrochemical Methods in Corrosion Research, Corrosion, 18, 338 (1962).

Patents:

Electrochemical Cells, April 1967, US 3,316,125.

Separation of Hydrogen by Permeation, November 1967, US 3,350,846.

Electrochemical Control of Sorption by Activated Charcoal, May 1973, US 3,730,885.

Process for Removing Hydrogen Sulfide and Ammonia from Gaseous Streams, March 1980, US 4,192,854, and corresponding Japanese Patent No. 1446500 registered June 30, 1988.

Removing Carbon Oxysulfide from Gas Streams, November 1981, US 4,298,584 and corresponding foreign patents.